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(71) Applicant

Katashi Aoki,  
6037 Ohazaminamijo, Sakakimachi, Hanishina-gun,  
Nagano-ken, Japan

(72) Inventor

Katashi Aoki

(74) Agent and/or address for service

Forrester, Ketley & Co., Forrester House, 52 Bounds  
Green Road, London, N11 2EY

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(58) Field of search

B5A

## (54) Method of making a hollow article of polypropylene by biaxial stretch blow moulding

(57) A method is disclosed of manufacturing, by biaxial stretch blow moulding, a hollow article of polypropylene, such as a bottle which is transparent and has luster and a uniform wall thickness as a whole.

In the method, fully mixed and blended molten polypropylene is injected into a parison mould to form a parison, and the parison is removed from the parison mould as soon as possible, whilst only a surface layer has cooled sufficiently to form a skin. The parison is transferred immediately to the cavity of a temperature control device, where the parison is subjected briefly to a preliminary stretching and blowing to expand it into contact with the surface of the cavity. The parison is then allowed to contract again and is transferred to a blow mould where it is subjected to a conventional stretch blow moulding process to produce the desired hollow article.

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## SPECIFICATION

**Method of Making a Hollow Article of Polypropylene by Biaxial Stretch Blow Moulding**

5 Polypropylene is a crystalline resin, and is generally supplied to manufacturers of mouldings and the like in the form of white crystalline pellets. The temperature at which polypropylene may be moulded is in the range from 190° to 260°, and it is desirable that where hollow articles such as bottles are moulded by blow moulding, injection blow moulding or the like, the temperature is set to a level as high as possible.

10 In blow moulding or injection moulding, since a moulded parison is immediately blow moulded into a hollow article, the transparency of hollow articles obtained is degraded and the articles are whitened or made somewhat opaque.

15 To make hollow polypropylene articles which are transparent, biaxial stretch blow moulding, a technique generally used to obtain transparent hollow articles of polyethyleneterephthalate, may be employed.

20 However, when moulding of a polypropylene hollow article is carried out by the biaxial stretch blow moulding technique in which a parison formed by injection moulding is stretched and blow moulded in a single operation, i.e. using what is generally referred to as the "hot parison system", even if the temperature of the parison is adjusted to the level most suitable for moulding, the parison is likely to be damaged during the process of stretching and blow moulding, and even if a usable hollow article should be obtained, the hollow article is uneven in wall thickness and is irregular in transparency, so that the product is generally unacceptable.

25 It is believed that the above-described non-transparency of the hollow article results from crystals formed during cooling of the resin after the melting thereof, and differences in transparency arise from differences in the dimensions of the crystals formed and the degree of crystallisation in the otherwise amorphous material. Accordingly, if the entire process from injection moulding of the parison to completion of the stretch blow moulding of the hollow article is carried out in an extremely short period of time, a hollow article which is excellent in transparency and has a uniform wall thickness might be obtained. However, apparatus generally used at present is unable to achieve this, and it has not yet been possible to manufacture hollow articles of polypropylene, which are transparent and have luster and a uniform wall thickness, by biaxial orientation.

30 It is an object of this invention to provide a method of making a hollow article of polypropylene, by biaxial stretch blow moulding, whereby an aesthetically acceptable article such as a bottle of polypropylene which is transparent and has luster and a uniform wall thickness as a whole, may be produced.

According to the invention, there is provided a

65 method of making a hollow article of polypropylene by biaxial stretch blow moulding, the method comprising: fully blending plasticised polypropylene within an injection cylinder by rotation of an injection screw until all material in a crystalline state has been converted to an amorphous state filling a parison mould with said plasticised polypropylene to mould a parison; releasing said parison from the mould, while the parison, apart from a thin skin layer formed on the surface of the parison, is still at high temperature and in an amorphous state, stretching and expanding said parison within a temperature control mould in a cavity in said temperature control mould which is larger than the parison as formed in said parison mould, expanding the parison by application of pressure internally thereof to bring its outer surface into close contact with the inner surface of the temperature control mould to adjust the parison temperature; thereafter removing said pressure to allow the parison to shrink from said inner surface of the temperature control mould, and then stretch blow moulding the parison within a blow mould to obtain a transparent hollow moulded article of polypropylene which is biaxially stretched.

70 Generally, in prior practice, pellets of plastics stock material are plastified by rotation of an injection screw within a heated injection cylinder, and the molten plastics material accumulating at the front end of the injection cylinder is injected into the mould by the forward movement of the injection screw. In normal injection moulding practice the pellets are intended to be completely molten by the rotation of the injection screw, by the time that the charging phase is completed, i.e. before injection moulding commences.

75 Crystalline resin such as propylene has an amorphous phase and a crystalline phase and both of these phases may be present in one both of the material, depending on the degree of melting, and even if the material is sufficiently molten for injection moulding of a parison to be possible, there is sometimes present a quantity of the material in the crystalline phase. It has been found that as long as the molten polypropylene includes material in the crystalline phase, a biaxially oriented hollow article which is transparent and has luster may not be obtained by stretching and blowing a parison moulded from the material.

80 It seems that the charge of material at the front end of the injection cylinder has irregularities in temperature even if all of the material is in the molten state. These irregularities of temperature still persist when the molten material is injected into the mould, and this is one of the causes of an irregularity of temperature in the parison. The unevenness of parison temperature can be corrected and the parison temperature rendered more uniform by the control of temperature by placing the parison in a cavity in a temperature control device maintained at a desired temperature, prior to the carrying out of the stretch blow moulding step. However, whilst such

correction can effectively correct external temperature irregularities resulting from the cooled parison mould, it is difficult to make, by such temperature control during the short period of time normally available, correction also of internal temperature irregularities already present in the molten plastics material prior to moulding the parison. The aforesaid irregularities in temperature cause differences in cooling speed of different portions of the parison when stretch blow moulding takes place, so that some portions are easily stretched and some portions are not easily stretched, and as a result, a defective article is produced in which irregularities in transparency and wall thicknesses are present.

In accordance with this invention, injection moulding of a parison is carried out after the polypropylene has been fully blended, i.e. after the amorphous material and the crystalline material in the charge of polypropylene in the injection cylinder have been mixed by rotation of an injection screw to allow the temperature of the charge of material to be rendered even. This blending ensures that the crystalline material is converted to the amorphous phase before injection takes place and at the same time, the evenness in temperature of the charge minimises unevenness of internal temperature of the parison once moulded. In fact, temperatures throughout a parison moulded after such blending has been carried out and throughout a parison moulded in the normal way have been measured, and it has been found that the former parison has less pronounced irregularities in temperature than the latter, and has better transparency than the latter, the latter being materially whitened as compared with the former.

In preferred embodiments of this invention, in moulding a parison, injection time and cooling time are made as short as possible and the parison released from the mould after merely such time as is necessary for sufficient cooling to form only thin skins on the inner and outer surfaces of the parison.

In the parison thus removed from the mould, the polypropylene may be partially crystallised in the thin skin layer on the inner and outer surfaces of the parison so that these skin layers become whitened. However, the plastics material remains at relatively high temperature and in the amorphous state in the region between said thin skins and the thin skins of the inner and outer surface are quickly re-melted by heat retained in said region, so that the parison gradually becomes transparent once more.

A hot parison released from the mould is adjusted in temperature to a moulding temperature by a temperature control device of the heat exchanging type. Such temperature control is carried out by the means disclosed in my U.K. Patent Application No. 8320096, and is carried out within the cavity in the temperature control device. The cavity in the temperature control device is of such a size as to be

substantially larger than the parison as the latter leaves the parison mould and initially there is a substantial clearance between the outer surface of the parison, and internal surface of said cavity. However, once located in the cavity, the parison is stretched axially and air under pressure is supplied to the interior of the parison so that the parison is expanded and its outer surface is thereby brought into close contact with the inner surface of said cavity to adjust the temperature of the parison to the stretch blow moulding temperature. The parison is axially stretched to the required dimension prior to the aforesaid expansion, and an appreciable part of the aforesaid temperature adjustment takes place in the time during which the parison contracts after the internal pressure in the parison is removed and the means used to stretch the parison axially is withdrawn.

To provide sufficient stretching and expansion, the inner surface area of the temperature control member is formed larger than the outer surface area of parison. However, if the difference between said inner and outer surface areas is excessively great, the length and diameter of the parison may be excessively permanently increased before the stretch blow moulding step so that the stretching and expansion which the parison undergoes in the stretch blow moulding stage decreases, with consequent failure to achieve sufficiently the desired biaxial orientation of the plastics.

Accordingly, there is a limit to the extent of stretching and expansion of the parison which may be effected within the temperature control device, and preferably, the inner surface area of the temperature control device cavity is from 1.4 to 2 times the outer surface area of the parison as the latter leaves the parison mould.

The aforesaid stretching of the parison in the temperature control device is carried out by inserting a stretching rod into the parison, and the expansion is carried out by blowing air into it. By the stretching and blowing, the outer surface of the parison is brought into close contact with the inner surface of the temperature control device to effect immediate heat-exchange, during which period the air pressure remains applied.

The time required for this temperature control is preferably within the time at which when the parison reaches its stretching blow moulding temperature and when the internal pressure is removed, the parison contracts. This contraction greatly affects the parison. Because the wall thickness of the parison increases as a whole during such contraction, an equilibration of heat is effected during the variation thereof whereby the temperature of the parison is made uniform and simultaneously, residual stress generated at the time of injection moulding is removed. Furthermore, because the parisons, when the internal pressure is removed, is moved away from the internal surface of the temperature control device, heating of the parison is automatically stopped at that stage.

The parison with its temperature thus adjusted, is immediately transferred to a blow mould and is axially stretched by a rod within the blow mould and expanded in the blow mould by blowing air into the parison, to fill the blow mould. The product within the mould cools rapidly and the moulded product has excellent transparency.

Various examples of the manufacture of products by a method embodying the invention are set out below. In these examples, moulding was carried out by an injection blow moulding machine such as disclosed in U.S. Patent No. 4,105,391.

#### EXAMPLE 1

Polypropylene in pellet form was heated, melted and fully blended by rotation of an injection screw within an injection cylinder heated to 220°C, and thereafter injected into a mould cooled to a temperature of 12° to 15°C to mould a bottomed hot parison. The polypropylene material used was as follows:—

Polypropylene Mitsubishi Chemical Industries Ltd. 6200E

MFI 1.7 g/10 min.

Density 0.897 g/cm<sup>3</sup>

Melting temp. 140°

The dimensions of the parison were as follows:—

Outside diameter 22 mm, Inside diameter 15 mm, Wall thickness 3.5 mm, Overall length 128 mm, Length below neck 110 mm.

The aforesaid hot parison was removed from the mould while its temperature was as high as possible, whilst still being in a releasable state and was transferred to a heat-exchanging type temperature control device for immediate temperature adjustment. At this stage, the temperature of the hot parison was 100° to 103°C. The temperature adjustment was carried out under the following conditions:

Temperature control device

Cavity inside diameter 36 mm

Cavity depth 137 mm

Temperature 120 to 130°C

Time 6 to 7 sec.

Air pressure 1 to 2 Kg/cm<sup>2</sup>

The temperature adjustment was carried out first by inserting the stretching rod into the hot parison, axially stretching the hot parison to the bottom surface of the cavity in the temperature control device, blowing air into the hot parison to expand the latter, and bringing the outer surface of the hot parison into close contact with the inner surface of the temperature control device.

The temperature adjustment was completed by the shrinkage of the parison which occurred when the stretching rod pressing the parison and the air pressure were removed.

The dimensions of the parison after temperature adjustment were as follows:—

Outside diameter 31 mm, length below neck 130 mm. The temperature of the parison was 113° to 120°C.

The aforesaid parison after the temperature adjustment steps, was immediately transferred to a position within a blow mould and stretch blow moulded by conventional means into a 600 ml Beier bottle. At this stage, pressure utilized was 8 to 10 Kg/cm<sup>2</sup>, and time for stretch blow moulding was 3 to 5.5 sec.

The dimensions of the bottle were as follows:—

Outside diameter of bottle (ellipse) 80x60 mm, wall thickness 0.4 mm, overall length 198 mm, length below neck 180 mm, weight 22.2 g.

The above described moulded article was transparent without whiteness, and had luster and uniform wall thickness.

#### EXAMPLE 2

An oil bottle of 700 ml was moulded by a method corresponding to that of Example 1 with the only differences being those indicated in the data set out below:—

Dimension of parison

Outside dia. 28 mm, inside dia. 20.6 mm, wall thickness 3.7 mm, overall length 130 mm, length below neck 118 mm

Temperature of parison mould 15 to 22°C

Temperature of parison 100°C

Temperature control device

Cavity Inside dia. 40 mm, depth 122 mm, temperature 90° to 100°

air pressure 8 Kg/cm<sup>2</sup> temperature adjustment time 0.5 to 1 sec.

Dimension of temperature adjusted parison

Outside diameter 33 mm, length below neck 119 mm temperature 115°C

Oil bottle (moulded article)

Outside diameter  $\phi$ 70 mm (Max  $\phi$ 77 mm)

Overall length 225 mm, wall thickness 0.4 mm, length below neck 213 mm, weight 29.6 g.

The above described moulded article also had excellent transparency and had luster and uniform wall thickness.

The described method embodying the invention for producing a hollow article in biaxially stretched polypropylene has the further advantage that it can be carried out using apparatus conventionally used for the production, by injection and stretch blow moulding, of articles in polyethylene terephthalate and the like.

#### CLAIMS

1. A method of making a hollow article of polypropylene by biaxial stretch blow moulding, the method comprising: fully blending plasticised polypropylene within an injection cylinder by

- rotation of an injection screw until all material in a crystalline state has been converted to an amorphous state filling a parison mould with said plasticised polypropylene to mould a parison;
- 5 releasing said parison from the mould, while the parison, apart from a thin skin layer formed on the surface of the parison, is still at high temperature and in an amorphous state, stretching and expanding said parison within a temperature
- 10 control mould in a cavity in said temperature control mould which is larger than the parison as formed in said parison mould, expanding the parison by application of pressure internally thereof to bring its outer surface into close contact with
- 15 the inner surface of the temperature control mould to adjust the parison temperature; thereafter removing said pressure to allow the parison to shrink from said inner surface of the temperature control mould, and then stretch blow
- 20 moulding the parison within a blow mould to obtain a transparent hollow moulded article of polypropylene which is biaxially stretched.
2. A method according to claim 1 wherein the parison temperature is in the range of 100° to
- 25 103°C, temperature control is carried out for about 0.5 to 7 sec. within the temperature control mould at 90° to 130°C.
3. A method according to claim 1 or claim 2 wherein the inner surface area of the temperature
- 30 control mould is from 1.4 to 2 times greater than the outer surface area of the parison as formed in said parison mould.
4. A method of making a hollow article of polypropylene by biaxial stretch blow moulding,
- 35 substantially as hereinbefore described.
5. A hollow article of polypropylene, made by the method of any of claims 1 to 4.